भारतीय मानक
Indian Standard

IS 5182 (Part 19) : 2022

वायु प्रदूषण मापने की पद्धतियाँ

भाग 19 क्लोरीन

(पहला पुनरीक्षण)

Methods for Measurement of Air Pollution

Part 19 Chlorine

(First Revision)

ICS 13.040.20

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BUREAU OF INDIAN STANDARDS
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FOREWORD

'This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.'

Chlorine gas is primarily a respiratory irritant. It is so irritating that concentrations above 9 mg/m^3 in air are readily detectable by a normal person. Chlorine is a very reactive element and reacts with other chemicals (both inorganic and organic/bio-organic molecules) present in environment, water bodies, earth's crust and the resultant molecules have different profile and affect the flora and fauna in different manners.

Devices and techniques for determining the concentration of pollutants in the atmosphere are important for the assessment of 'ambient air quality', establishing hazardous levels in the environment, adoption of ameliorative measures and appraisal of contamination from a process or source.

The standard was originally published in 1982. Based on the technological development in this area since last three decades, the committee decided to revise the standard.

In this revision, the following changes have been made:

a) References have been updated;

b) Preparation of stock chlorine solution has been modified;

c) Sampling period has been specified; and

d) Calculation has been revised.

In the preparation of the original standard, considerable assistance was derived from Publication No. 42215-01-70 'Tentative methods of analysis for free chlorine content of the atmosphere (methyl orange method)' issued by the American Public Health Association. In the development of this first revision, considerable assistance has been taken from Methods of air sampling and analysis (Third Edition) – James P. Lodge, Jr., Method No. 202.

The composition of the technical committee responsible for formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 19 CHLORINE

(First Revision)

1 SCOPE

This standard (Part 19) describes methyl orange method for measurement of free chlorine in air.

2 REFERENCES

The following Indian Standard contains provisions which through reference in this text, constitutes provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
5182 (Part 5) : 1975	Methods for measurement of air pollution: Part 5 Sampling of gaseous pollutants

3 METHYL ORANGE METHOD

3.1 Principle

The colour of methyl orange solution ceases to vary with acidity near a *p*H value of 3.0. The dye is quantitatively bleached by free chlorine, and the extent of bleaching may be determined colorimetrically. The optimum concentration range is 0.05 to1.0 ppm in ambient air (145 µg/m^3 to 2900 µg/m^3 at 25 °C and 760 mm Hg).

NOTE — It is essential to maintain proper *p*H.

3.2 Range and Sensitivity

The procedure given is designed to cover the range of 5 to 100 µg of free chlorine per 100 ml of sampling solution. For a 30 l air sample, this corresponds to approximately 0.05 to 1.0 ppm in air which is the optimum range. Increasing the volume of air samples extends the range at the lower end, but only within limits, since 50 l of chlorine-free air produces the same effect as about 0.01 ppm of chlorine.

3.3 Interferences

Free bromine, which gives the same reaction, interferes in a positive direction. Manganese (III, IV) in concentrations of 0.1 ppm or above also interferes positively. Nitrites impart an off-colour orange to the methyl orange reagent. Nitrogen dioxide interferes positively, reacting as 20 percent chlorine. Negative interference from sulphur dioxide is significant both in solution as well as gaseous state, decreasing the effect due to chlorine by an amount equal to one third the sulphur dioxide concentration.

3.4 Reagents

Pure chemicals of analytical grade, unless otherwise specified, and distilled water (*see* IS 1070) shall be used in the test. These shall not contain any impurities that affect the results of analysis.

3.4.1 *Methyl Orange Stock Solution,* 0.05 *percent*

Dissolve 0.5 gm of methyl orange in distilled water and dilute to 1 l. This solution is stable indefinitely if freshly boiled and cooled distilled water is used.

3.4.2 *Methyl Orange Reagent,* 0.005 *percent*

Dilute 100 ml of the methyl orange stock solution (3.4.1) to 1 l with distilled water. Prepare fresh for use.

3.4.3 *Hydrochloric Acid Solution,* 5 N.

The 42 ml concentrated hydrochloric acid is diluted to 100 ml with distilled water.

3.4.4 *Sampling Solution*

Dilute 6 ml of 0.005 percent methyl orange reagent **(3.4.2)** to 100 ml with water and add 3 drops of 5 N hydrochloric acid. 1 drop of butanol may be added to induce foaming and increase collection efficiency. *p*H of the solution may be checked using a *p*H meter.

3.4.5 *Acidified Water*

Add 3 drops of 5 N hydrochloric acid to 100 ml of water.

3.4.6 *Potassium Dichromate Solution (0.1 N)*

Dissolve 4.904 gm anhydrous potassium dichromate primary standard grade in distilled water and dilute to 1 l.

3.4.7 *Starch Indicator Solution*

Prepare a thin paste of 1 gm of soluble starch in a few ml of water. Bring 200 ml of water to boil, remove from

heat, and stir in the starch paste. Prepare fresh before use.

3.4.8 *Potassium Iodide*, Reagent grade.

3.4.9 *Sodium Thio-sulphate Solution ($Na_2S_2O_3.5\ H_2O$),* 0.1N.

Dissolve 25 gm. of $Na_2S_2O_3.5\ H_2O$ in freshly boiled and cooled water and dilute to 1 l. Add 5 ml chloroform as preservative and allow to age for two weeks before standardizing. Take 80 ml water, 1 ml concentrated sulphuric acid and 1 gm of potassium iodide in a 500 ml conical flask and pipette 10 ml of 0.1 N potassium dichromate solution into the flask with constant stirring. Allow to stand in the dark for 6 min. Titrate with 0.1 N sodium thio-sulphate solution. Upon approaching the end point (brown colour changing to light yellowish green), add 1 ml of starch indicator solution and continue titrating to the end-point (blue to colourless). Note the titre value. Determine the strength of the sodium thio-sulphate solution, 0.1 N from the normality equation.

3.4.10 *Sodium Thio-sulphate Solution*, 0.01 N.

Dilute 100 ml of the aged and standardized 0.1 N sodium thio-sulphate solution (**3.4.9**) to 1 lwith freshly boiled and cooled distilled water. Add 5 ml of chloroform as preservative and store in a glass-stoppered bottle. Standardize frequently with 0.01 N potassium dichromate solution.

3.4.11 *Chlorine Solution,10 ppm*

Take sodium hypochlorite solution 4 percent *w*/*v* (that is, 40 000 ppm) as the stock chlorine solution. Prepare 500 ml of approximately 10 ppm chlorine solution by serial dilution of the stock NaOCl solution (4 percent *w*/*v*).

3.5 Apparatus

3.5.1 *Spectrophotometer*, Suitable for measurement at 505 nm, preferably accommodating 5 cm cells.

3.5.2 *Sampling Train*

According to IS 5182 (Part 5) using a large impinger with fritted disc [*see* Fig. 2 of IS 5182 (Part 5)] having 70 to 100 µm maximum pore diameter.

3.6 Sampling

Assemble the sampling train. Add 100 ml of sampling solution (**3.4.4**) to the impinger and draw a measured volume of air at a rate of 1 to 2 l/min for 1 to 4 h depending on the estimated chlorine concentration. Preserve the solution by covering the impinger with opaque paper to protect the solution from sunlight. The volume of sampling solution, the concentration of methyl orange in the sampling solution, the amount of air sampled, the size of impinger and the length of the spectrophotometer cell may be varied to suit the needs of the situation as long as proper attention is paid to the corresponding changes necessary in the calibration procedure.

3.7 Analysis

Transfer the impinger contents to a 100 ml volumetric flask and make up the volume, if necessary, with acidified water **(3.4.5)**. Measure the absorbance at 505 nm in 5 cm cells against distilled water as reference.

Also measure the absorbance of the unexposed sampling solution (blank) against distilled water as reference.

The actual absorbance of the sample is obtained by subtracting the absorbance of the unexposed sampling.

Solution (blank) from the observed absorbance of the sample.

3.8 Calibration

3.8.1 Prepare a series of six l00 ml volumetric flasks containing 6 ml of 0.005 percent methyl orange reagent (3.4.2), 75 ml distilled water and 3 drops of 5 N hydrochloric acid. Carefully and accurately pipette 0.5, 1.0, 3.0, 5.0 and 9.0 ml of chlorine solution (**3.4.11**) into the respective flasks holding the pipette tip beneath the surface. Quickly mix and make up the volume with distilled water. Prepare a reagent blank by taking 6 ml 0.005 percent methyl orange reagent, 75 ml. distilled water and 3 drops of 5 N hydrochloric acid in a 100 ml. volumetric flask and making up the volume with distilled water.

3.8.2 Immediately standardize the 10 ppm (approx.) chlorine solution (**3.4.11**) by adding 400 ml of the chlorine solution to a flask containing 1 gm potassium iodide and 5 ml glacial acetic acid and swirl to mix. Titrate with0.01 N sodium thio-sulphate (**3.4.10**) until the colour of solution becomes a faint yellow. Add 1 ml of starch indicator solution and continue the titration to the end-point (blue to colourless). Note the titre value. 1 ml of 0.01 N sodium thio-sulphate is equivalent to 0.3546 mg of free chlorine. Compute the amounts of free chlorine added to each flask (**3.8.1**) in mg.

3.8.3 Transfer the standard chlorine solutions as prepared in **3.8.1** to absorbance cells and take absorbance readings of the chlorine standards and reagent blank with distilled water as reference at 505 nm. Subtract the absorbance of the reagent blank from that of the standard solutions to get the actual absorbance values of the standard solutions. Plot the actual absorbance readings of the standard solutions *versus* the mg of free chlorine contained in the chlorine standards. Determine the slope of the curve.

3.9 Calculation

Calculate the chlorine concentration as follows:

The free Cl_2 content in the sample,in $\mu g/m^3$ =

$$\frac{A \times k \times 10^6}{V} \quad \ldots(1)$$

where

A = corrected absorbance of sample (observed abs. of sample – abs. of reagent blank) with respect to distilled water;

k = calibration factor = reciprocal of slope of calibration curve; and

V = volume of air sampled, l.

3.10 Effect on Storage

The colour of sampled solutions is stable for at least 24 hr if protected from direct sunlight, although the presence of certain interferences (Fe III) may cause slow colour change.

3.11 Precision and Accuracy

Error due to measurement by this procedure is known to be less than ± 5 percent of the amount present.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Air Quality Sectional Committee, CHD 35

Organization	*Representative(s)*
In Personal Capacity (Former Head, Environmental Testing and Analysis Division, BARC)	Dr Gauri Pandit (***Chairman***)
Avantha Centre for Industrial Research and Development, Yamuna Nagar	Dr Nishi Kant Bhardwaj Dr Sunil Kumar (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr A. Vinod Kumar Dr S. K. Sahu (*Alternate*)
Central Institute of Mining and Fuel Research, Dhanbad	Dr R. C. Tripathi Dr N. K. Srivastava (*Alternate*)
Central Pollution Control Board (MoEFCC), GoI, New Delhi	Shri V. K. Shukla Dr R. C. Srivastava (*Alternate*)
Confederation of Indian Industry, New Delhi	Shrimati Seema Arora Dr Nandini Kumar (*Alternate*)
Delhi Technological University (formerly known as Delhi College of Engineering), New Delhi	Dr Anubha Mandal
Directorate General, Factory Advice Service & Labour Institute, Mumbai	Dr Brij Mohan Shrimati Dhanashree Acharekar (*Alternate*)
Ecotech Instruments, Greater Noida	Dr Rajendra Prasad
Envirotech Instruments Pvt Ltd	Dr Balbir Singh Shri Ashish Gupta (*Alternate*)
Green Economy Initiatives Pvt Ltd, Mohali	Dr R. S. Saini
Gujarat Pollution Control Board	Dr D. N. Vansadia
Himachal Pradesh Pollution Control Board	Dr T. B. Singh Dr Manoj Chauhan (*Alternate*)
In personal capacity	Dr N. Raveendhar
In Personal Capacity (*1221, Mahatma Gandhi Road, P O, Haridevpur, Kolkata – 700 082*)	Shri Sanjib Kumar Goswami
In Personal Capacity (*H.No. 514, S S S T Nagar, Rajpura Road, Patiala 147 003 Punjab*)	Dr H. K. Parwana
In personal capacity	Dr S. K. Tyagi
Indian Association for Air Pollution Control	Dr J. S. Sharma
Indian Chemical Council, Mumbai	Dr Rakesh Kumar Dr Pranav K. Tripathi (*Alternate* I) Shri J. I. Sevak (*Alternate* II)
Indian Institute of Technology, Kanpur	Nomination Awaited
Indian Institute of Toxicology Research, Lucknow	Dr S. C. Barman
India Meteorological Department, New Delhi	Nomination Awaited
Jharkhand State Pollution Control Board, Ranchi	Shri R. N. Kashyap
Jindal Steel	Shri M. Vaidyanathan Dr S. Majumdar (*Alternate*)

Organization	*Representative(s)*
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Ministry of Environment and Forest	Nomination Awaited
National Environmental Engineering Research, Delhi	Dr S. K. Goyal Dr P. K. Labhasetwar (*Alternate*)
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Tata Consulting Engineers	Shri Srikant Patil Dr Jyoti Prabha (*Alternate*)
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The Fertilizer Association of India, New Delhi	Mr Manish Goswami
Thermofisher Scientific	Ms Jaya Nangia Shri Anant Ghadigaonkar (*Alternate*)
UNIPHOS Envirotronics Pvt Ltd, Gujarat	Shri Vijay Pandey Dr R. C. Naik (*Alternate*)
BIS Directorate General	Shri A. K. Lal, Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Ms Preeti Prabha
Scientist 'C' (CHD), BIS

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